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Surface Sampling for Contaminants Employing Infrared Spectroscopy

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ABSTRACT

This report covers a series of preliminary experiments that focus on the development of a quick and efficient method of sampling surfaces for toxic contaminants. An introduction outlines how the problem is approached, while an experimentation section details the procedures used to evaluate the feasibility of such a concept. The paper concludes by presenting the results and offering suggestions for the future development of a practical surface sampling procedure. Ideas for expanded features and applications of this new sampling procedure are also included. All tables and graphs referenced in this report may be found in Appendix A.

OBJECTIVE

Evaluate the potential of developing a simple yet sensitive sampling and analysis procedure for evaluating the state of contamination of structures and equipment in a post spill or chemical attack environment. Ideally, the surface sampling and analysis procedure will be quick and reliable, capable of being employed in rugged combat environments.

INTRODUCTION

The development of a simple surface sampling and analysis procedure appears to be a complex and multi-faceted problem. However, the need for a general and easy-to-understand procedure becomes greater every day in a number of disciplines. In this instance, a general surface sampling and analysis procedure will hopefully evolve into a front-line defense that protects forward-deployed servicemen and women in post spill or biochemical attack environments. In a combat situation, biochemical accidents and attacks must be assessed immediately to determine their impact on the mission. For this reason, a quick and easy surface evaluation procedure would be an invaluable tool, especially when the nearest professional chemical analyst may be hundreds of miles away. Though other applications of this procedure will be discussed towards the end of the paper, there will undoubtedly be far-reaching applications that cannot be envisioned all at once.

The development of a simple yet sensitive surface sampling and analysis procedure should employ infrared (IR) spectroscopy. Considering the procedure focuses on the detection of biochemical toxins, it must be designed to detect a wide variety of organic compounds, including organophosphorous nerve agents (e.g. Sarin, Soman, Tabun, and VX as documented on the first page of Appendix A), with a common procedure. Knowing that every organic compound possesses a unique IR spectrum, or so-called "fingerprint", IR spectroscopy would be an ideal approach to this problem. With comprehensive libraries of spectra already compiled on organic compounds, the spectrum of a surface sample could easily be cross-reference with a library of spectra using a computer comparison program to identify the contaminants. And even if a definite "fingerprint match" could not be made, the likelihood of toxic contamination could be quantitated by the computer based upon the similarities of the surface sample's spectrum to the spectra of known toxins.

Employing IR spectroscopy on the front-line, however, will require unique surface sampling and analysis techniques. Undoubtedly, a portable and rugged IR spectrometer and computer database will be needed to make the surface sampling procedure a reality, not to mention it should be user-friendly to even the most inexperienced of users. The analysis of surface samples, though, can only be achieved after a reliable procedure is developed for actually sampling surfaces. The selected surface sampling method must be compatible with almost any solid surface and not require painfully meticulous procedures. Remember that the primary application includes the chaotic, dirty, and desperate characteristics of a combat environment.

The first approach at a simple surface sampling procedure included the use of disposable IR cards. Handheld cards specially fit to an IR spectrometer, with a nearly IR-transparent polymer film over the card's aperture as a sample platform, seemed to be the ideal tool. The lack of availability of such disposable cards, however, prompted the brainstorming of a modified method. If an adhesive tape can be

developed with certain properties, then surface sampling would be greatly simplified. The adhesive tape could be applied to the surface to be evaluated for contamination, pressed down, and then peeled off. With the organic contaminants of the surface retained on the tape, the adhesive tape and the sample could be directly analyzed by an IR spectrometer. The spectrum collected would have the background of the adhesive tape subtracted out, so a computer comparison program could evaluate the spectrum of the surface contaminants. If the contaminants are identified by the computer comparison, then further analysis could be performed to determine the concentration of the contaminant, since a known area of the adhesive tape will have been applied to the contaminated surface. If not, a computer could easily provide a warning flag by quantitating the probability of toxic contamination.

Though the following experiment has considered many possible solutions to the problem at hand, it focuses specifically on the development of an adhesive tape for use in surface sampling. The concept of using an adhesive tape in surface sampling has unlimited potential. It can be developed so its applications span countless disciplines and its features provide many benefits.

EXPERIMENTAL PROCEDURES

The first step is to design a generic template for making blank IR cards, or cards that will fit into the Nicolet Magna 560 IR Spectrometer with an aperture at the necessary position. The template will be made to the appropriate size, so when it is used to trace an outline on a surface it will produce a card with the exact dimensions of the IR spectrometer's cardholder. The outlined cards must be two inches in width and four inches long, rounded on all corners. The thickness and durability of the cards can be tailored by selecting the right material. In this experiment, green government cardboard file-dividers will be used. The template can then be used to outline and cut as many blank IR cards as desired. Once the cards are cut out, the aperture can be made. The aperture should be no smaller than a ¾ inch diameter hole, centered laterally and 2½ inches from the bottom of the card. The second page of Appendix A illustrates the dimensions of the blank IR card. A drill press with a ¾ inch hole bit can then be used to make accurate apertures in the correct location. Simply ensure the desired number of blank IR cards are held together tightly, the center of the aperture is clearly marked on the top card, and drill through the entire stack of cards.

The IR spectra of several adhesive tapes will now be collected to determine if adhesive tapes in general are practical for use as sample platforms in infrared spectroscopy. Gather several different types of adhesive tape and ensure each one is uniquely labeled so its source is known. For all tapes, pull off a strip and place it over the aperture of one of the homemade IR cards. Be careful not to allow any foreign contaminants onto the part of the tape over the aperture, especially the adhesive side that cannot be easily cleaned, and including fingerprints. Collect the adhesive tape's spectrum with the IR spectrometer and subtract out the air background. Save the results, preferably in graphical form as a percent transmittance versus wavenumber graph.

Select the adhesive tape with the best percent transmittance across the IR spectrum. Ideally, a tape can be found that does not absorb any IR light. Since this is unrealistic, select the best tape by comparing their IR spectra. High transmittance across as much of the IR spectrum as possible is desirable. Peaks of no-transmittance should be sharp, few, and far-between. Ensure a region of the IR spectrum is not blocked that is necessary for identifying the contaminants of concern. In this experiment, the regions between 650 and 1500 cm^{-1} and 2500 and 3000 cm^{-1} are critical regions for identifying the spectra of organophosphorous nerve agents.

Now use the selected adhesive tape to determine if it is an adequate means of sampling for and analyzing your contaminant. Cover the aperture of the blank IR card with the adhesive tape. Place some of your contaminant, or a simulant of your contaminant if the contaminant you wish to identify is toxic, on a surface. Place the IR card over the surface with the adhesive side of the tape down, press the adhesive onto the surface, and pull the IR card off. Collect the spectrum of the IR card with the surface sample in the IR spectrometer. Be sure to subtract out the background of the adhesive tape. Note how the contaminant physically and chemically interacts with the adhesive tape.

Compare a known spectrum of the contaminant to the spectrum of the contaminant collected experimentally. Hopefully, the spectra are identical and a positive match can be made with the surface sampling procedure. If so, continue experimentation and attempt to sample your contaminant off of various types of surfaces, in smaller concentrations, and under variable conditions. This will test the flexibility and sensitivity of the sampling procedure. If the procedure was unsuccessful, further modifications will have to be made by identifying the problem and taking corrective actions. Every problem will require a different approach. Some may require an adjustment in the bench settings of the IR spectrometer, while others may require a specific sampling method or a completely different adhesive tape. If possible, attempt to identify the exact material of the adhesive tape that works best. Specific adhesive resins and polymer films may block certain parts of the IR spectrum, produce undesirable noise, or interact destructively with the contaminant.

RESULTS

The green homemade IR cards worked great in this experiment. Not only were they cheap and easily produced in mass, but also provided the perfect durability.

The adhesive tapes considered for use in the sampling procedure included household desk tape, yellow transparent tape from Bruce Nielsen's office, clear government-supply packaging tape, Saran Wrap, Glad Cling Wrap, Manco packaging tape, LePage transparent tape, yellow 3M Scotch Tape, 3M transparent note tabs, orange transparent tape from a lab drawer, and double-sided 3M Scotch Tape from the Fabrication Shop. The three selected for continued experimentation were the clear government-supply packaging tape, Glad Cling Wrap, and yellow 3M Scotch Tape. The percent transmittance graphs for the three adhesive tapes are found as pages 3, 4, and 5, respectively, in Appendix A. Note that the yellow 3M Scotch Tape is actually an orange color and carries a government identification number for supply purposes.

Four organic substances were chosen for the testing of the three adhesive tapes. Trinitrotoluene (TNT), naphthalene, acetophenone, and DMMP absorb IR light in areas close to those of organophosphorous nerve agents. DMMP is a nerve agent simulator. The absorbance spectra for naphthalene, acetophenone, and DMMP are found on pages 6, 9, and 11, respectively, in Appendix A.

50μL of 1000μg/mL solution of TNT was placed directly onto the adhesive side of the government packaging tape and the Glad Cling Wrap. The TNT appeared to have a phobia for the tape, for it "ran" away from the tape and was absorbed by the cardboard IR card. The spectrum of TNT was never successfully obtained with the sampling procedure.

Enough of a 0.100g/mL solution of Naphthalene dissolved in methylene chloride was poured to cover the bottom of a Pyrex beaker. The solution was allowed to sit until dry. The dried Pyrex surface with Naphthalene was then sampled with an IR card made with the government packaging tape, and its spectrum collected. The spectrum of naphthalene was identifiable, based upon the theoretical spectrum obtained from a library of organic spectra in Dr. Howard Mayfield's possession. However, there was a significant oscillating noise that confused the identification of the naphthalene spectrum (see page 7 of Appendix A for graphical results).

Glad Cling Wrap was tested in the same way as the packaging tape and naphthalene. It was slightly more difficult to get the surface sample onto the polymer film, since the Cling Wrap does not have the adhesive nature of the tapes. For this reason, the Glad Wrap was rubbed on the surface in order to get an adequate surface sampling. The naphthalene spectrum was detectable through the Glad Wrap. An oscillating noise was still present, though it was at a lower intensity than with the packaging tape.

Several procedures were attempted to eliminate the oscillating noises. The bench settings of the Magna IR 560 were adjusted to increase the aperture size. Though the relative size of the noise was reduced as the setting was increased, the oscillating noise remained significant. The government packaging tape and Glad Cling Wrap were also substantially stretched before surface sampling in the hopes a porous polymer film

would solve the oscillating noise problem. The noise did not go away. Spectra of the naphthalene on packaging tape were also collected with the Nicolet Magna IR 750 Spectrometer. In addition, the IR cards were turned at an acute angle to the IR beam in an attempt to reflect any noise away from the spectrometer's optical sensors. All spectra included the same oscillating noise.

Surface samples of naphthalene, acetophenone, and DMMP were all taken with the yellow 3M Scotch Tape (see pages 8, 10, and 12, respectively, in Appendix A). There was no oscillating noise and all spectra were clearly identifiable. As with all previous trials, these trials included a noise burst between 2800 and 3000 cm^{-1}, where the polymer of the adhesive tapes completely block IR light.

Further experimentation was performed to test the ability of the yellow 3M Scotch Tape to preserve samples of acetophenone. It is observed that acetophenone evaporates completely within a few hours when exposed to air. Surface samples were collected in the same way, but multiple spectra were collected over a period of twenty minutes for each sample. Over the twenty-minute period, the intensity of the acetophenone spectrum on a single layer of yellow 3M Scotch Tape decreased (see page 13 of Appendix A). When the sample was preserved by placing a second layer of tape over the sample (i.e. "sandwiching" the acetophenone between two layers of tape), there was no observable decrease in the intensity of the acetophenone spectrum (see page 14 of Appendix A).

DISCUSSION AND ANALYSIS

It seems there are a variety of adhesive tapes that are capable of sampling surfaces for organic compounds and then serving as platforms for infrared analysis. However, there are unique results and problems with each tape. In most cases, the selected tape is a sufficient platform and allows for the collection of identifiable spectra. This assumes the adhesive tape transmits infrared light with some consistency across the infrared spectrum. Even adhesive tapes that block up to 25 percent of the light across the entire infrared spectrum and leave some scattered "windows" of transmittance may serve as adequate adhesive tapes. Ideally, however, an adhesive tape with the infrared transparency of a disposable 3M IR Card is desired (see page 15 of Appendix A).

Currently, the yellow 3M Scotch Tape appears to be the best adhesive tape for this project. Though its transmittance of infrared light is not the greatest of the tapes, it still transmits enough infrared light that the spectra of our contaminants can be identified. The government packaging tape has a great potential, but more research will have to be performed to eliminate the oscillatory noise. Attenuated Total Reflectance is a possible solution, and may reveal better procedures for the analysis of surface samples on adhesive tapes. All polymer tapes appear to have noise problems in the regions they completely absorb infrared light.

Stretching the government packaging tape and Glad Cling Wrap appeared to have no effect on the spectra collected. Further research, however, should be conducted to reveal if a porous polymer has any advantages over the traditional polymer films used in adhesive tapes.

The yellow 3M Scotch Tape successfully preserved a sample of acetophenone. Though the experimentation was brief, it made a definite point. More conclusive results will require testing over a longer period of time, with different organic compounds, and under various conditions.

CONCLUSIONS

There is a great potential for the use of an adhesive tape to develop a simple yet sensitive surface sampling and analysis procedure. The experiment proves an adhesive tape can be used to sample surfaces for organic contaminants, directly analyze them though infrared spectroscopy, and identify the contaminants by the collected spectra. Additionally, a sampling technique that employs a dependable adhesive tape offers an easy way to gather samples that anyone can understand. The discovery or creation of the ideal adhesive tape would undoubtedly lead to the ideal surface sampling and analysis technique. This experiment has demonstrated the feasibility of the concept with little effort, and leaves much room for further refining of the procedure. This technique, however, will not be a success unless a rugged, portable, and compatible infrared spectrometer complements it, ideally with a computer system capable of analyzing the collected spectra. The product will then become a common, yet highly important piece of equipment throughout the armed services.

FUTURE RESEARCH AND DEVELOPMENT

Though great things have been revealed in the initial five weeks of experimentation, there remains much ground to cover. The following list describes the avenues for future research and development that will make this project a success:

The thorough research of adhesive tapes, ending in the selection of the ideal materials for the best adhesive tape for this project, including the exact polymer film and adhesive resin. Desirable properties include maximum infrared transmittance, especially in the regions where organophosphorous nerve agents register, reduced noise when analyzed with a contaminant by infrared spectroscopy, adhesiveness with organic compounds, and no reactivity with contaminants.

A study on the chemical interactions between contaminants and adhesive tapes that may ruin sampling.

The analysis of porous polymers for their ability to retain samples and possibly reduce noise.

Noise problems studied and minimized, possibly using different tapes and alternative methods of infrared spectroscopy (e.g. Attenuated Total Reflectance).

Research performed on the types of contaminants that can or cannot be detected with this technique, and which types of surfaces or combinations of contaminant and surface can be successfully sampled.

A determination of the smallest concentration or amount of contaminant that can be detected with this technique.

A study performed on the extent to which surface samples may be preserved with the idea of "sandwiching" the sample between two layers of adhesive tape.

The development or discovery of a compatible infrared spectrometer that is rugged and user-friendly. Keep in mind that the adhesive tape procedure can be modified to some degree to match existing spectrometer characteristics.

POTENTIAL APPLICATIONS

Undoubtedly, the applications of a simple yet sensitive surface sampling and analysis procedure are innumerable. While the product can be specifically tailored for the identification of organophosphorous nerve agents on various surfaces in forward-deployed bases, the potential of a general product is unlimited. Any number of agencies could use the product on any number of surfaces while looking for any type of contaminant. The product may even evolve into a common device used in laboratories throughout the world. Health agencies may find it convenient for collecting and analyzing surfaces during inspections, while criminal investigators may find it useful for collecting and preserving samples of organic evidence. Even medical facilities could use it to sample possible victims' skin for contaminants. Also, the information gathered in this research may be applied to the development of a biochemical warfare shelter.

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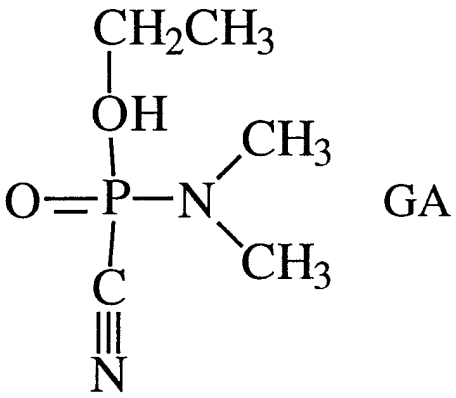
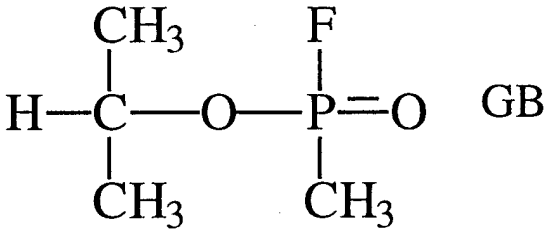
Appendix A

Organophosphorus Nerve Agents



GA

Dimethylphosphoramidocyanidic acid, ethyl ester, Sarin



GB

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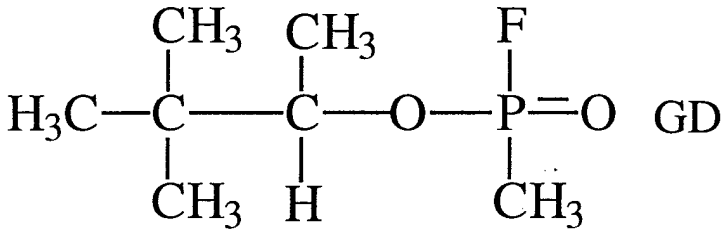
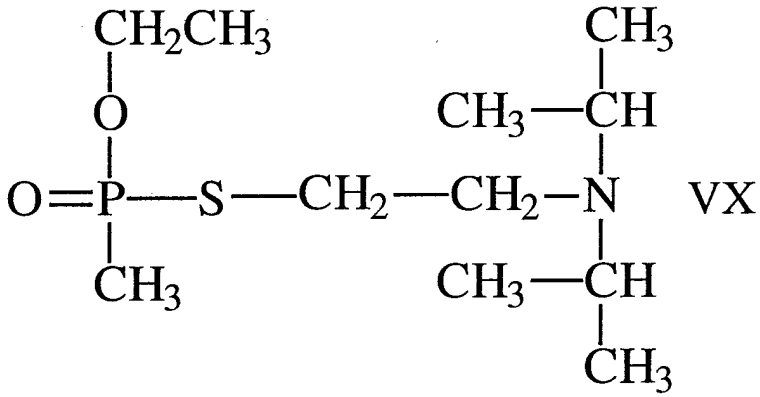
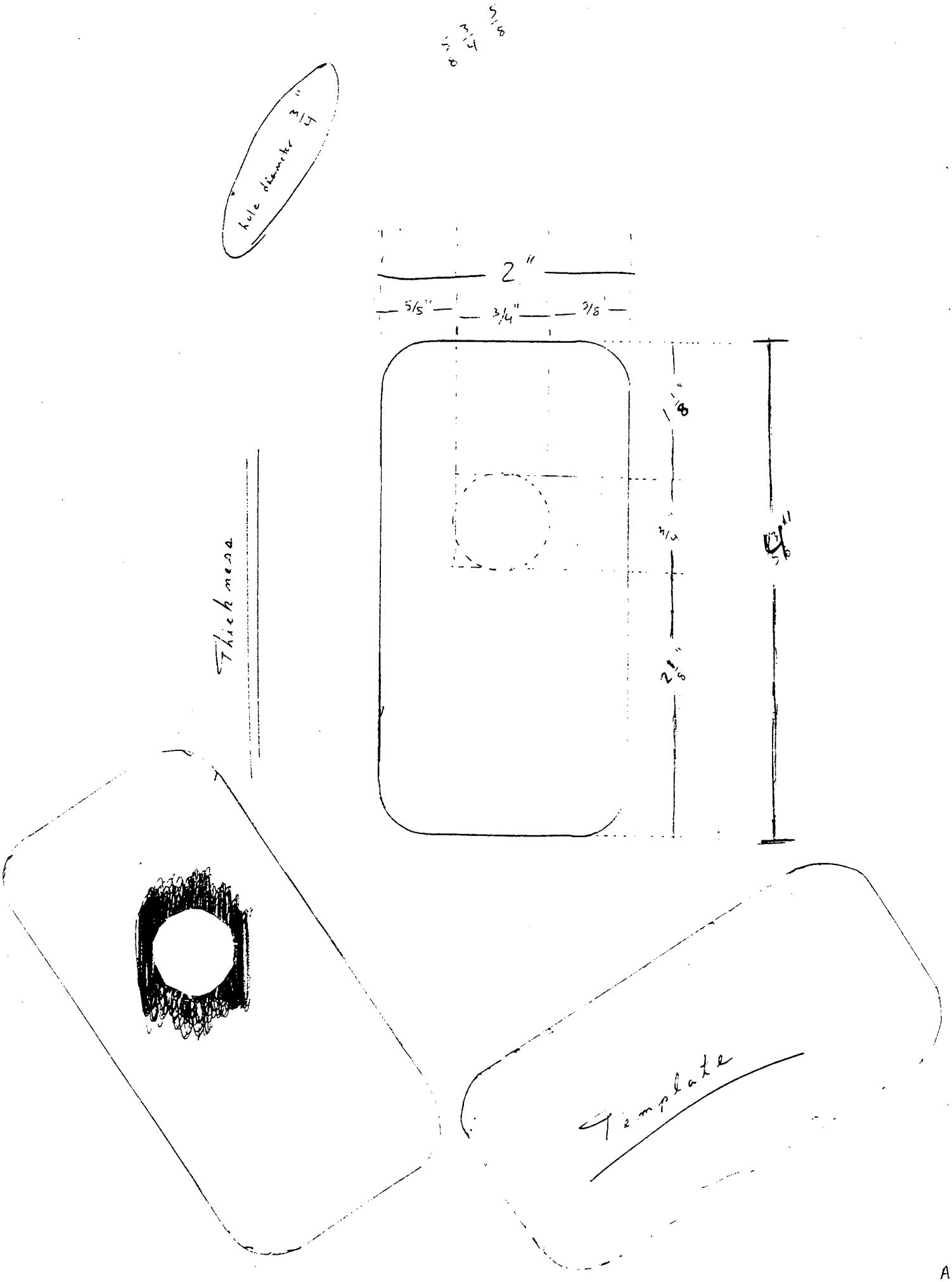


GD

Methylphosphonofluoridic acid, 1,2,2-trimethylpropyl ester, Tabun



Methylphosphonothioic acid, S-[2-[bis(1-methylethyl)amino]ethyl] O-ethyl ester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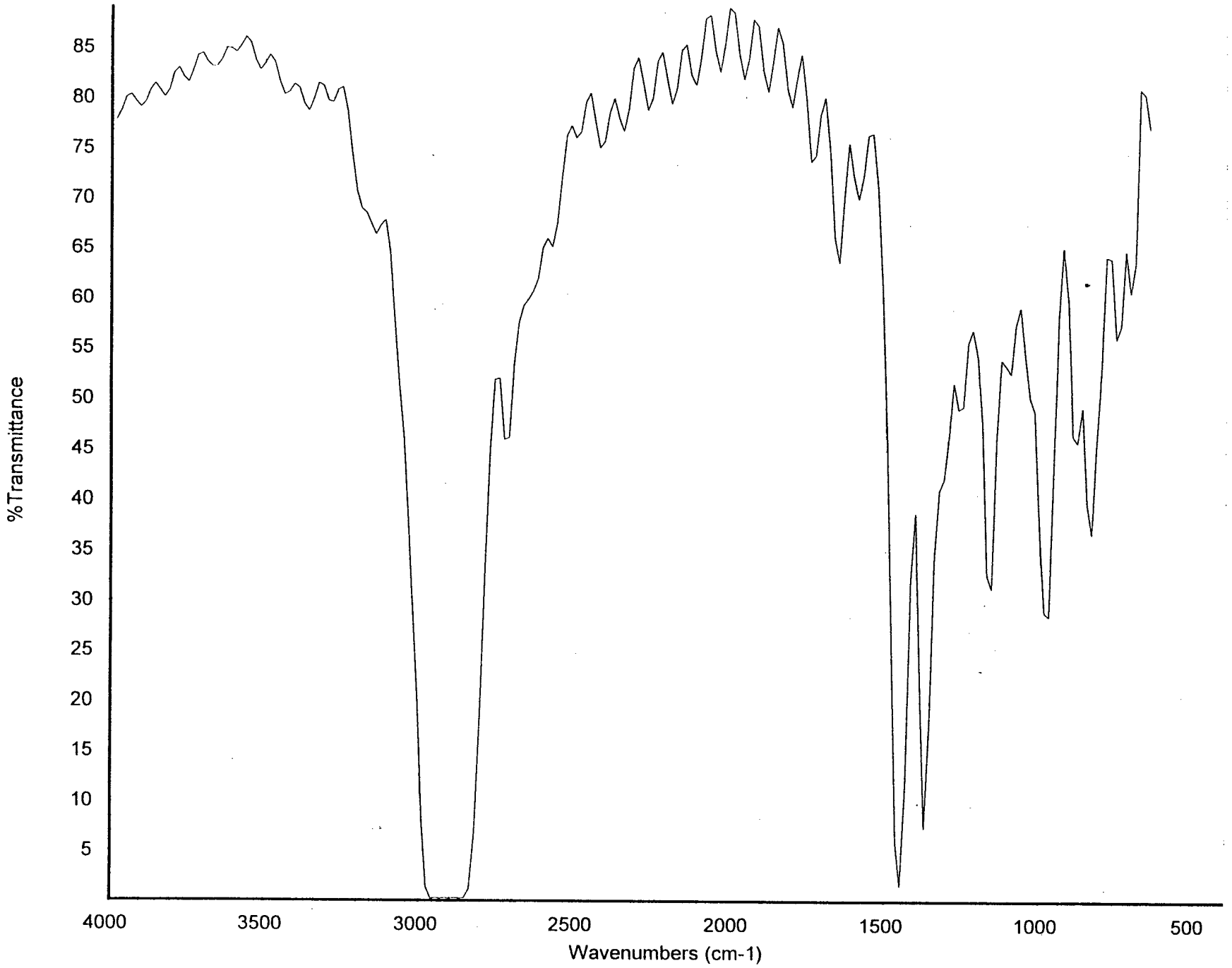
3/4"
5/8"
1/8"
2 1/8"
Thickness
Template



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Fri May 28 09:43:03 1999 Packaging Tape

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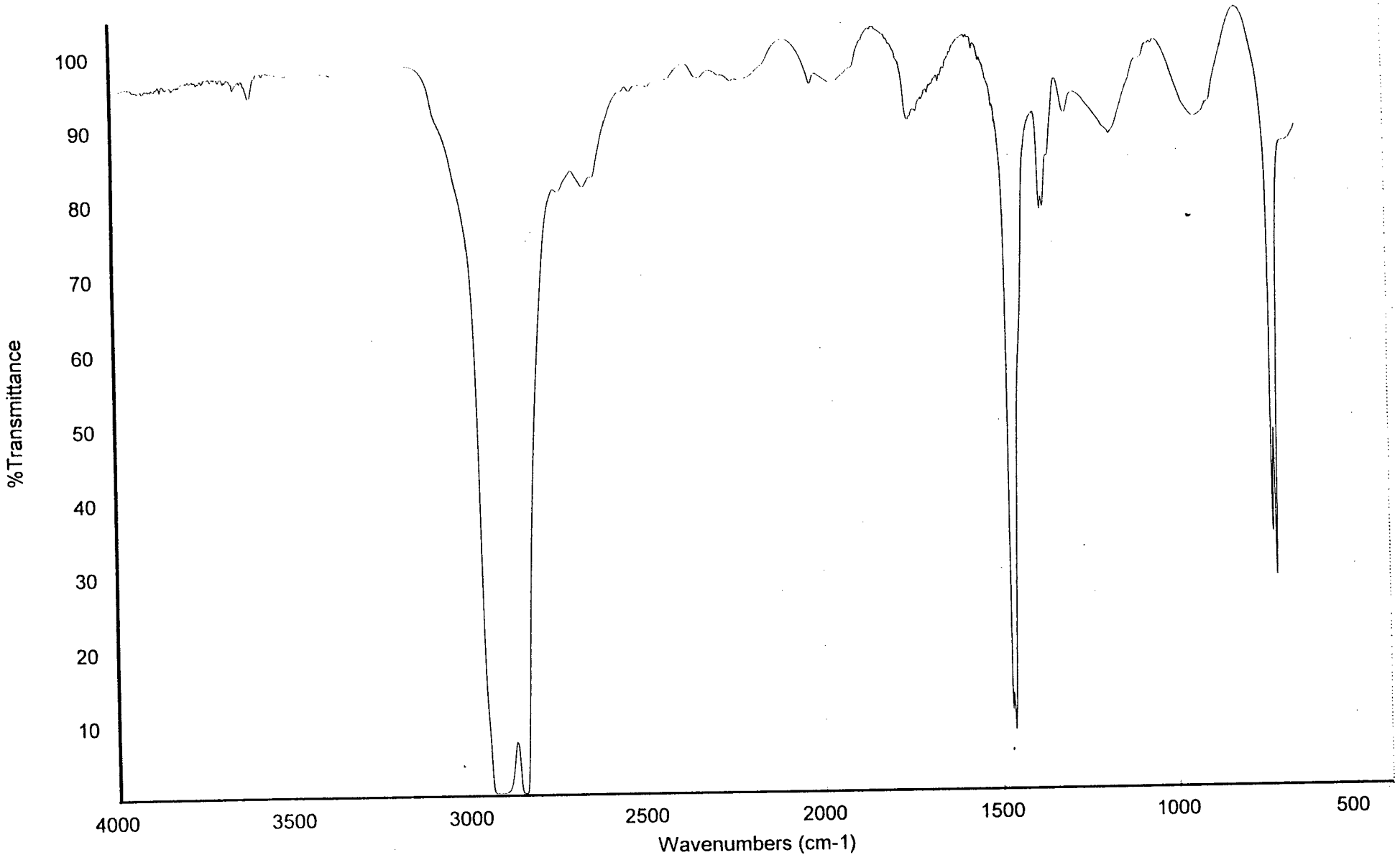
A3



Glad Cling Wrap

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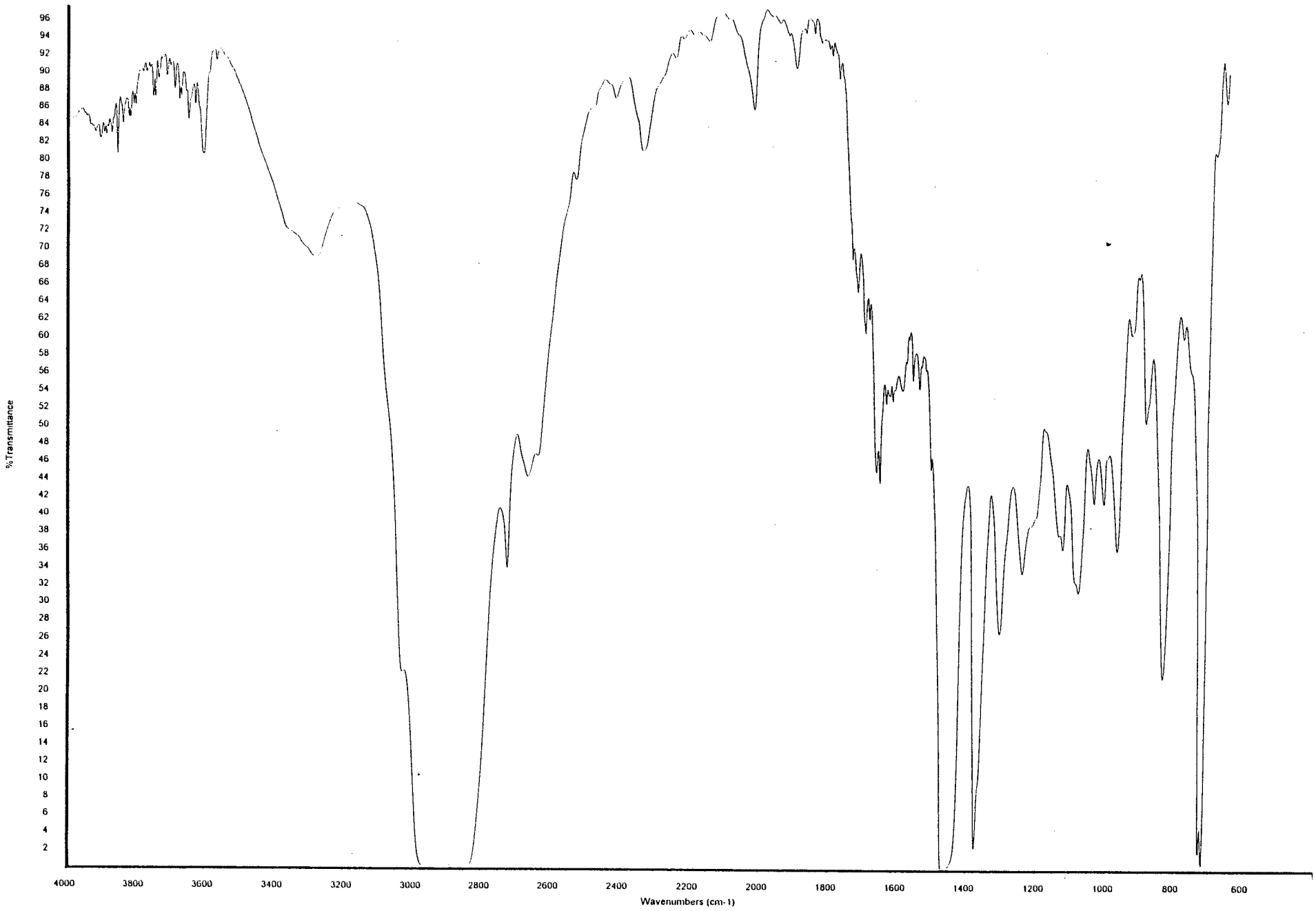
A4



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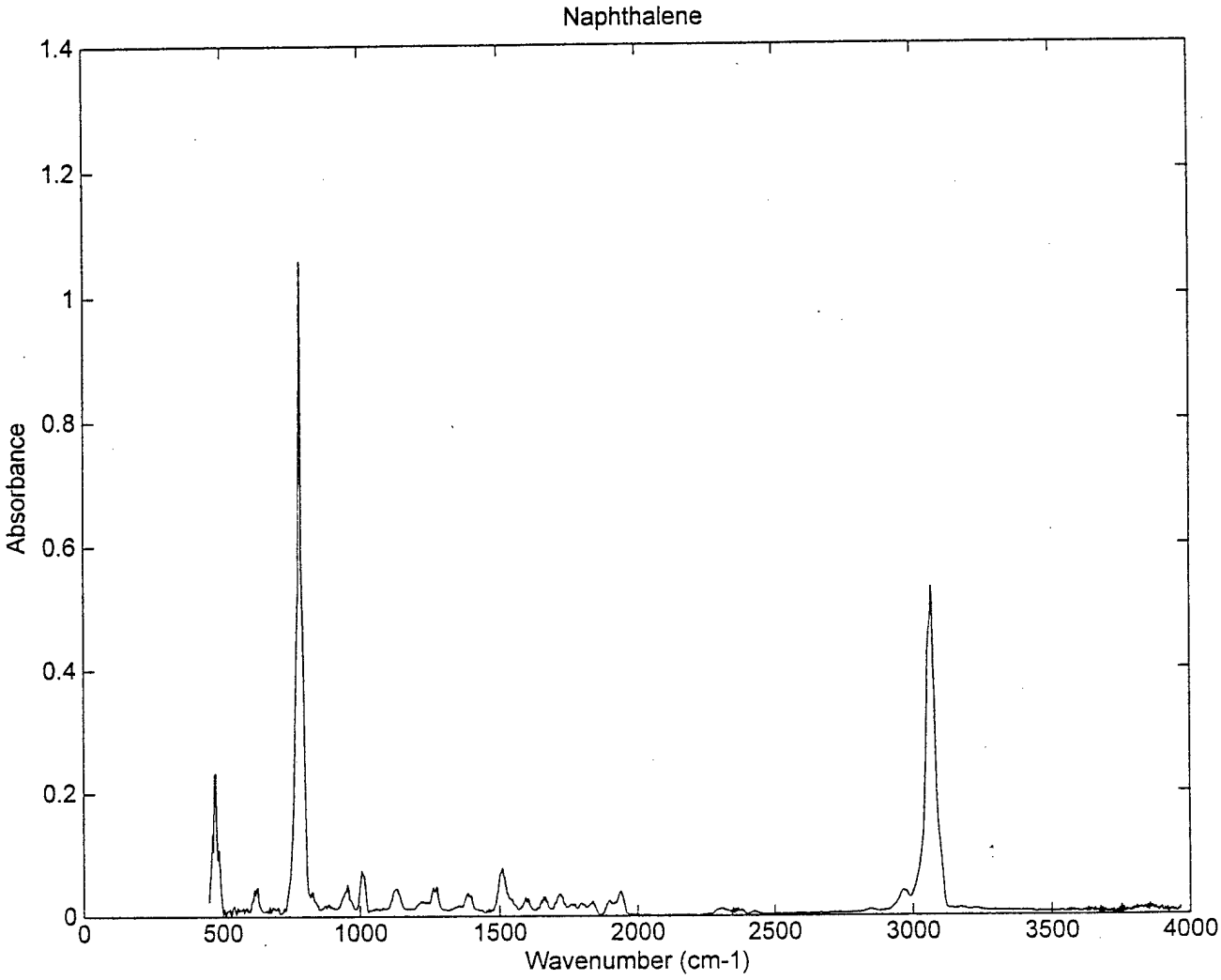
Yellow 3M Scotch Tape from Lab

Scans 8

Resolution 4.000

A5


Naphthalene
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0.8
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1.2
1.4
0
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1000
1500
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2500
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3500
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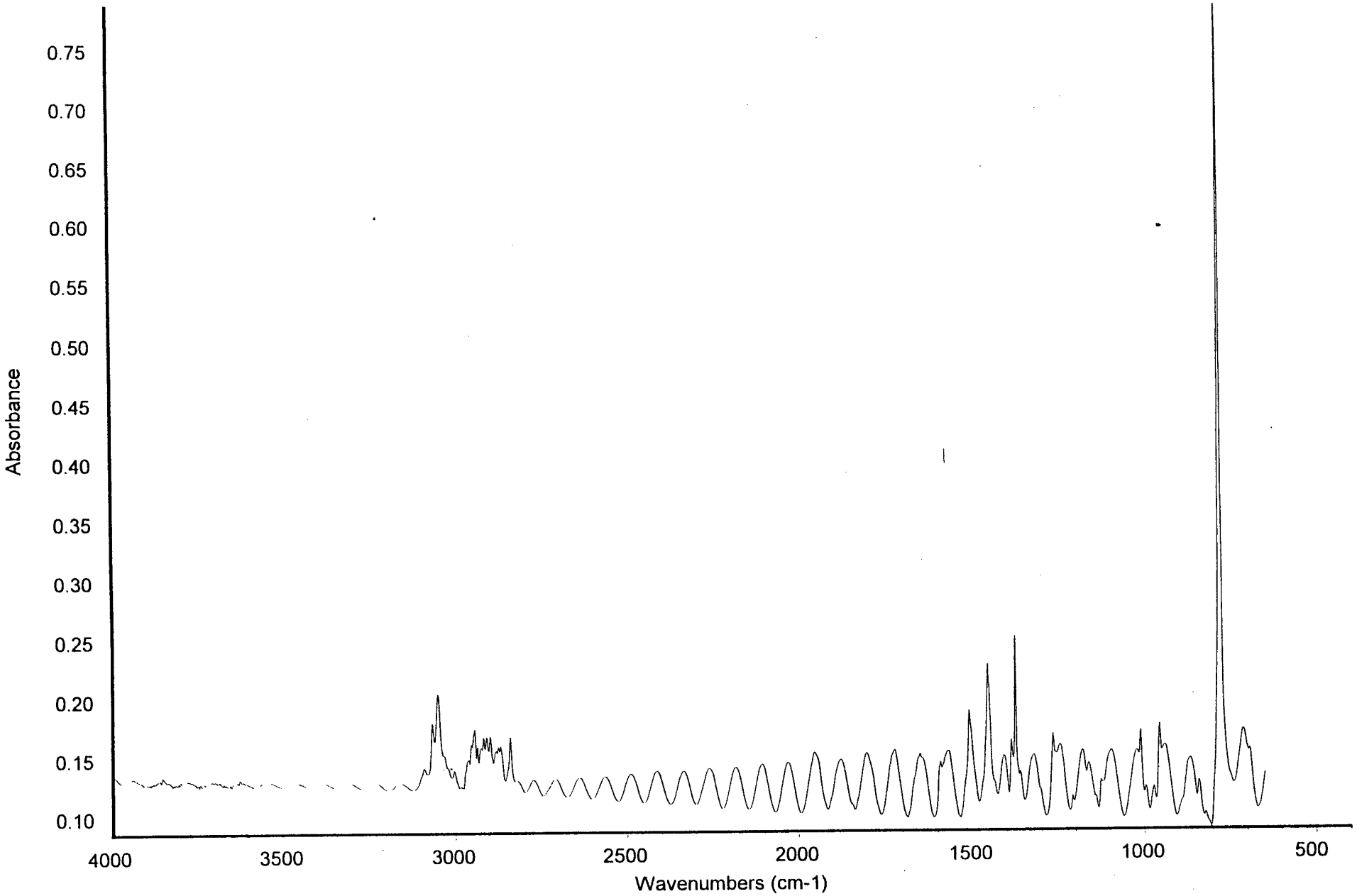

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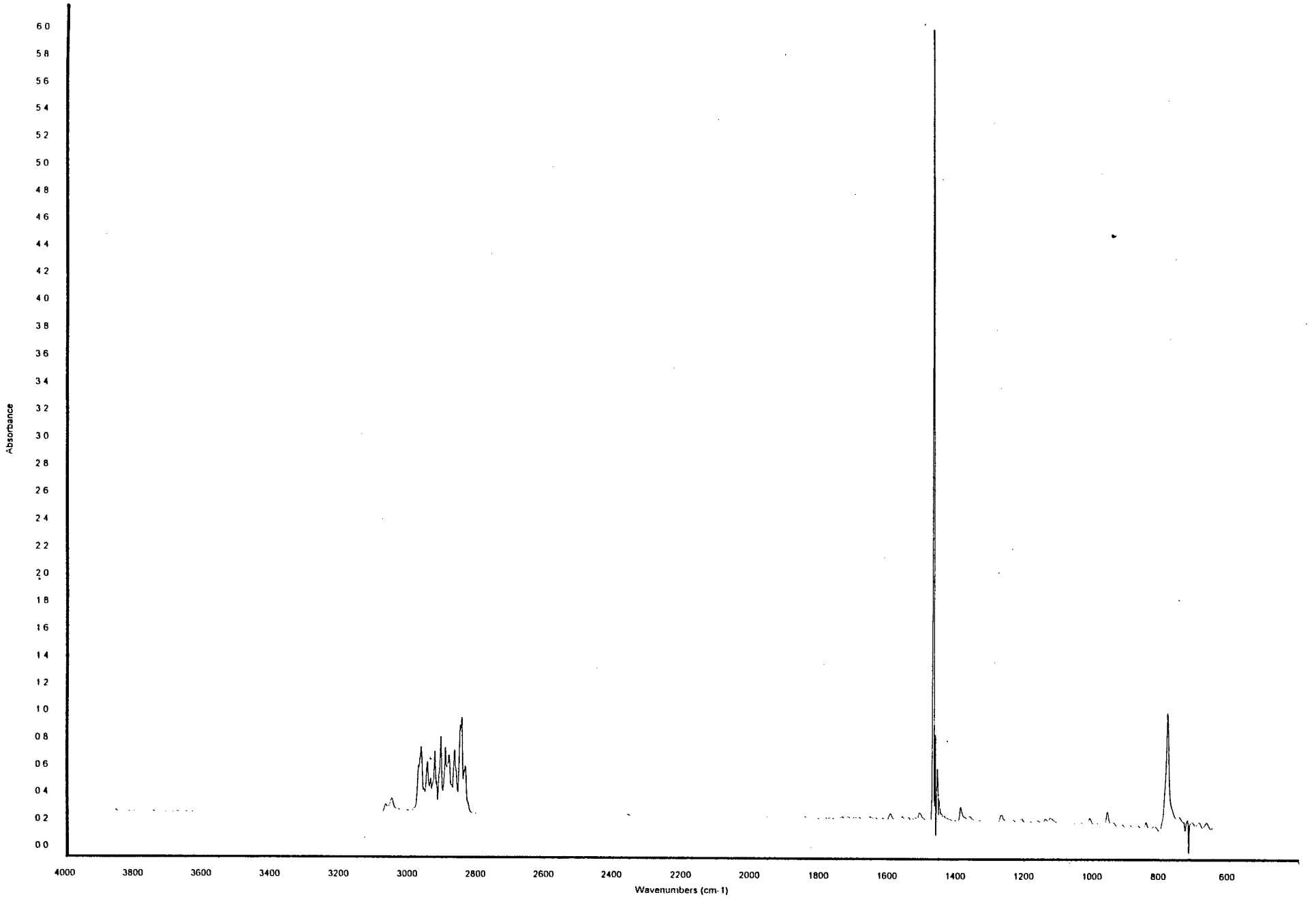
Card #2

Gen't packaging tape w/ naphthalene

A7



Date Fri Jun 18 09 50 11 1999

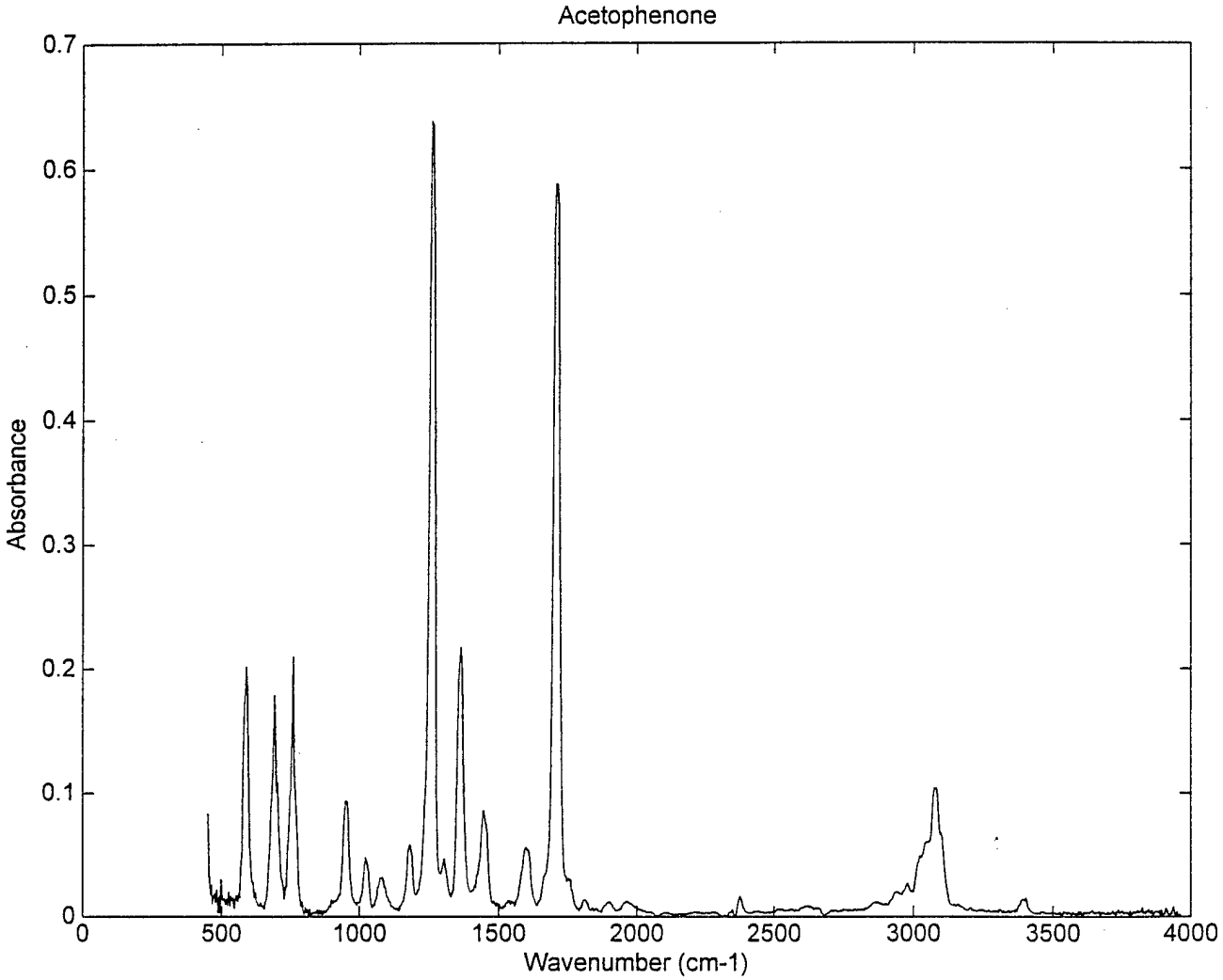
Yellow 3M Scotch w/ Naphthalene

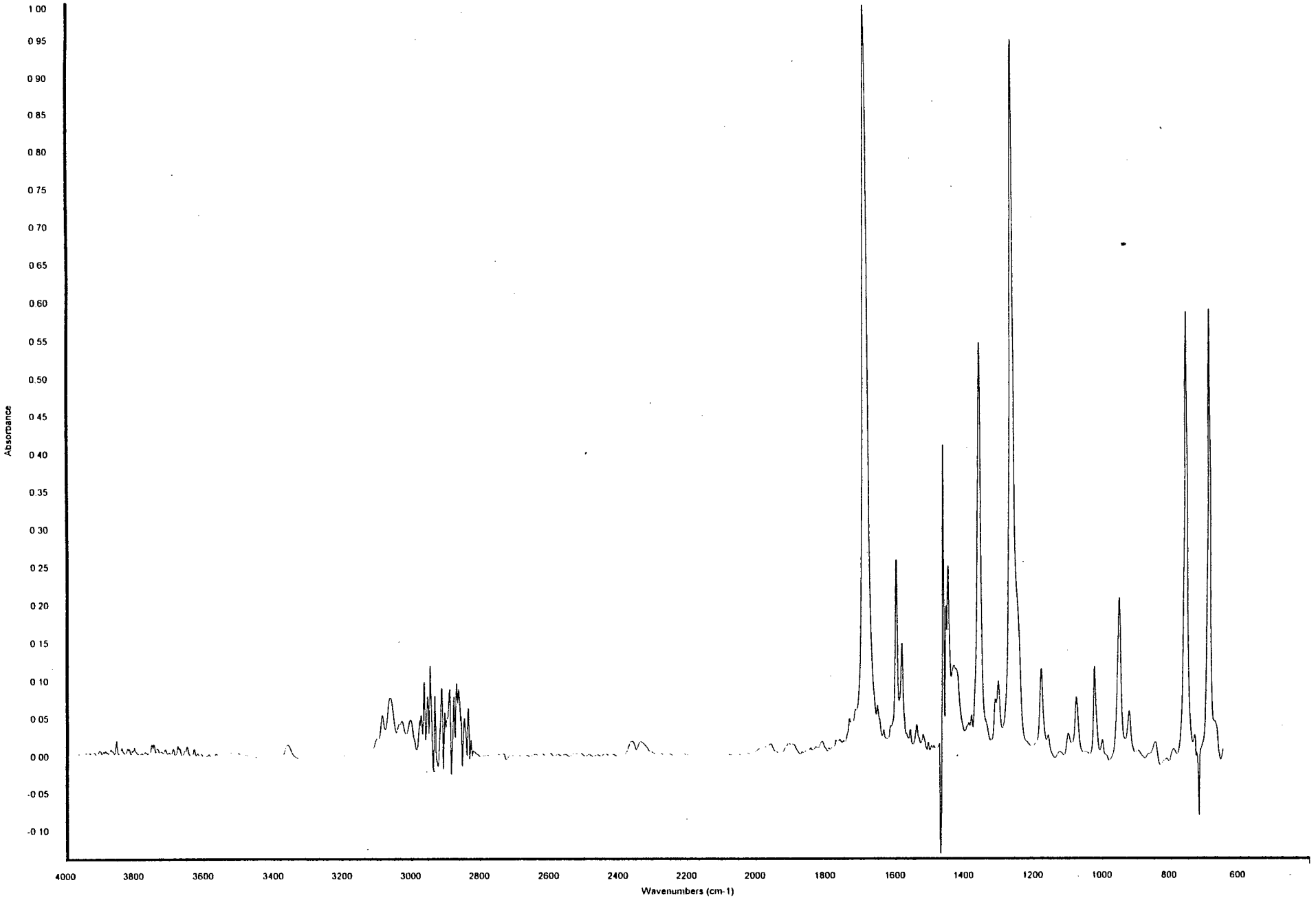
Scans 8

Resolution 4 000

A8

CS 0-821


Acetophenone
Absorbance
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
500
1000
1500
2000
2500
3000
3500
4000
Wavenumber (cm-1)



Date Fri Jun 18 10 45 03 1999

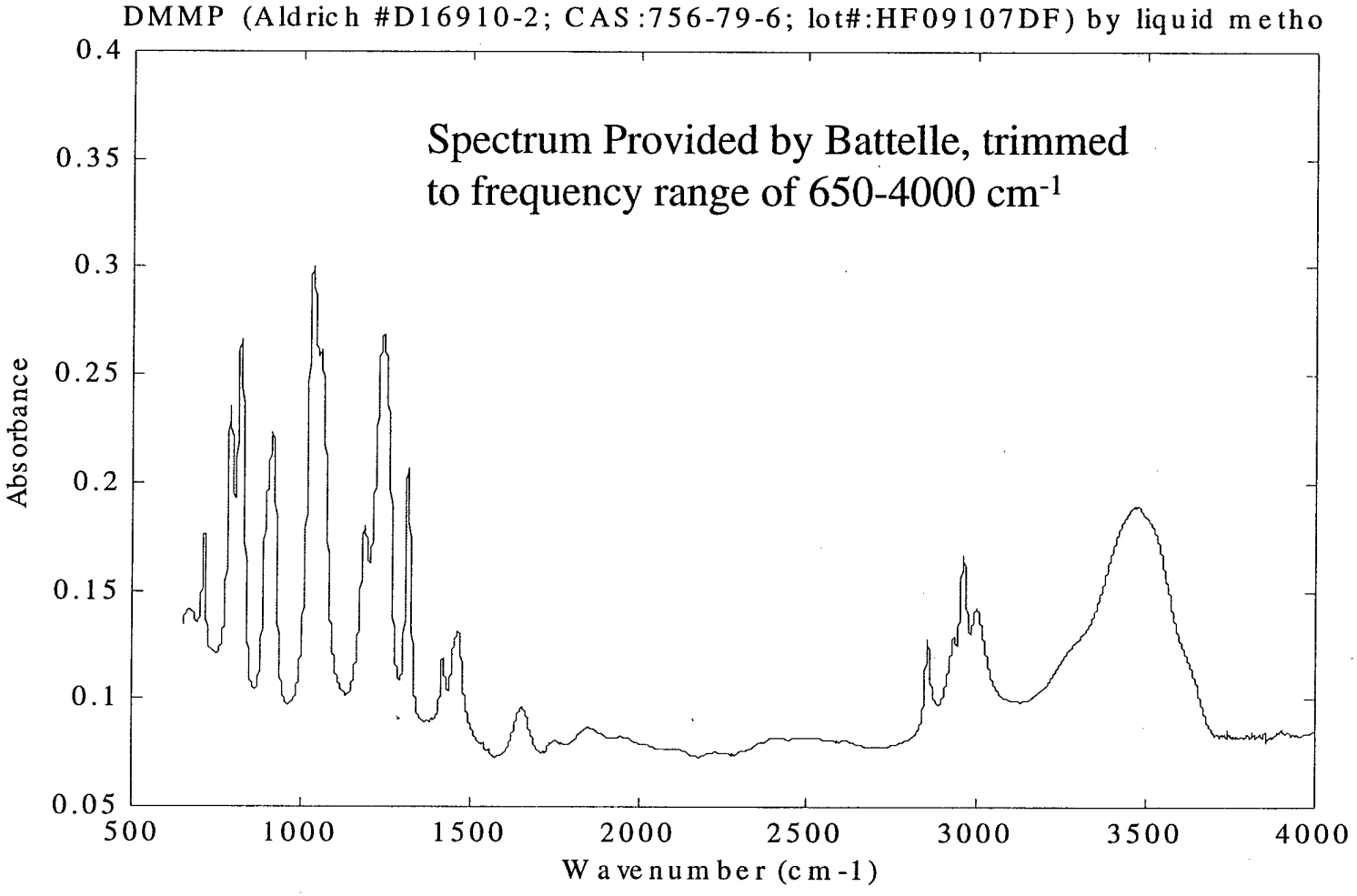
Yellow 3M Scotch w/ Acetophenone picked up off beaker

Scans 8

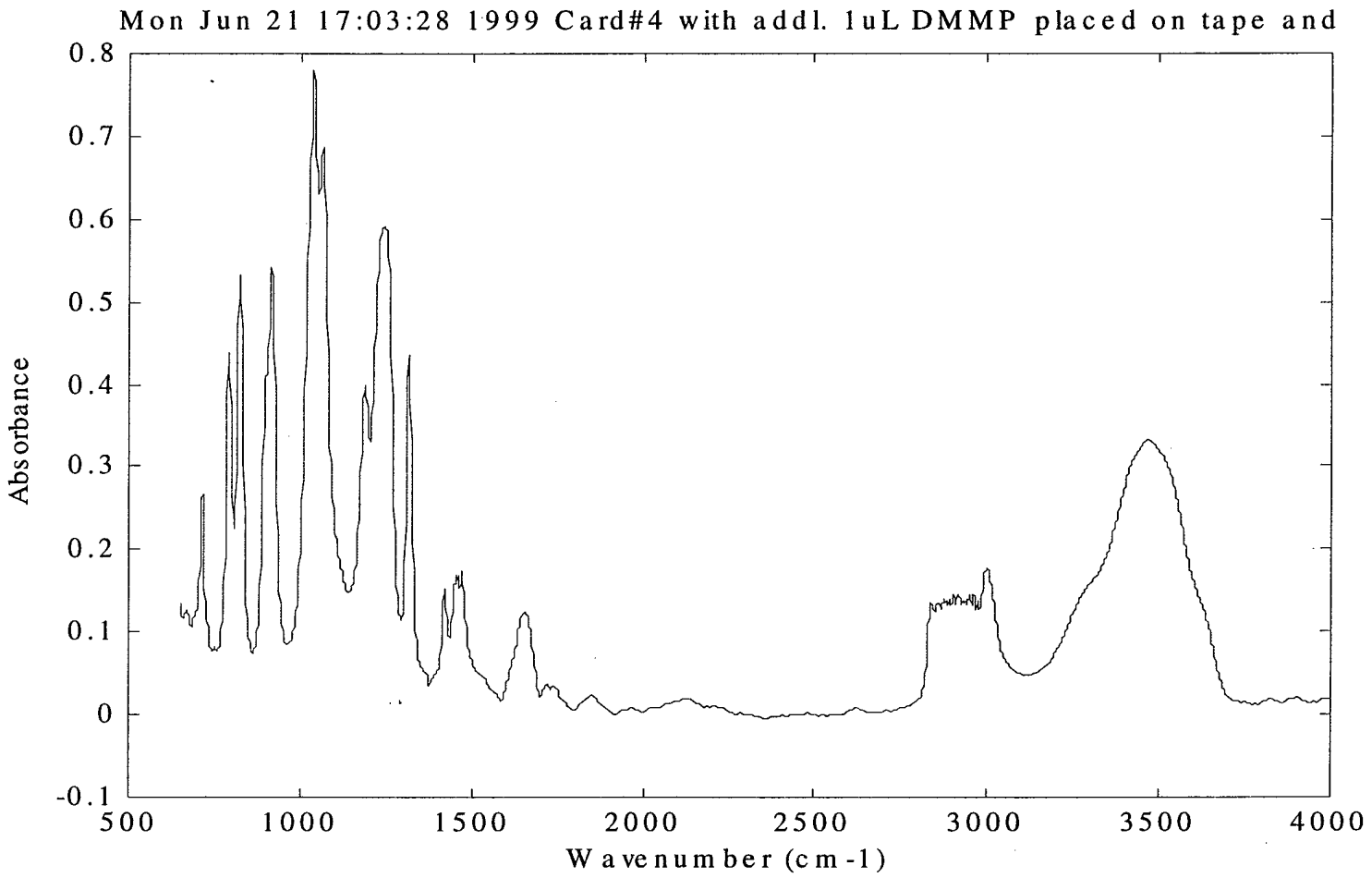
Resolution 4 000

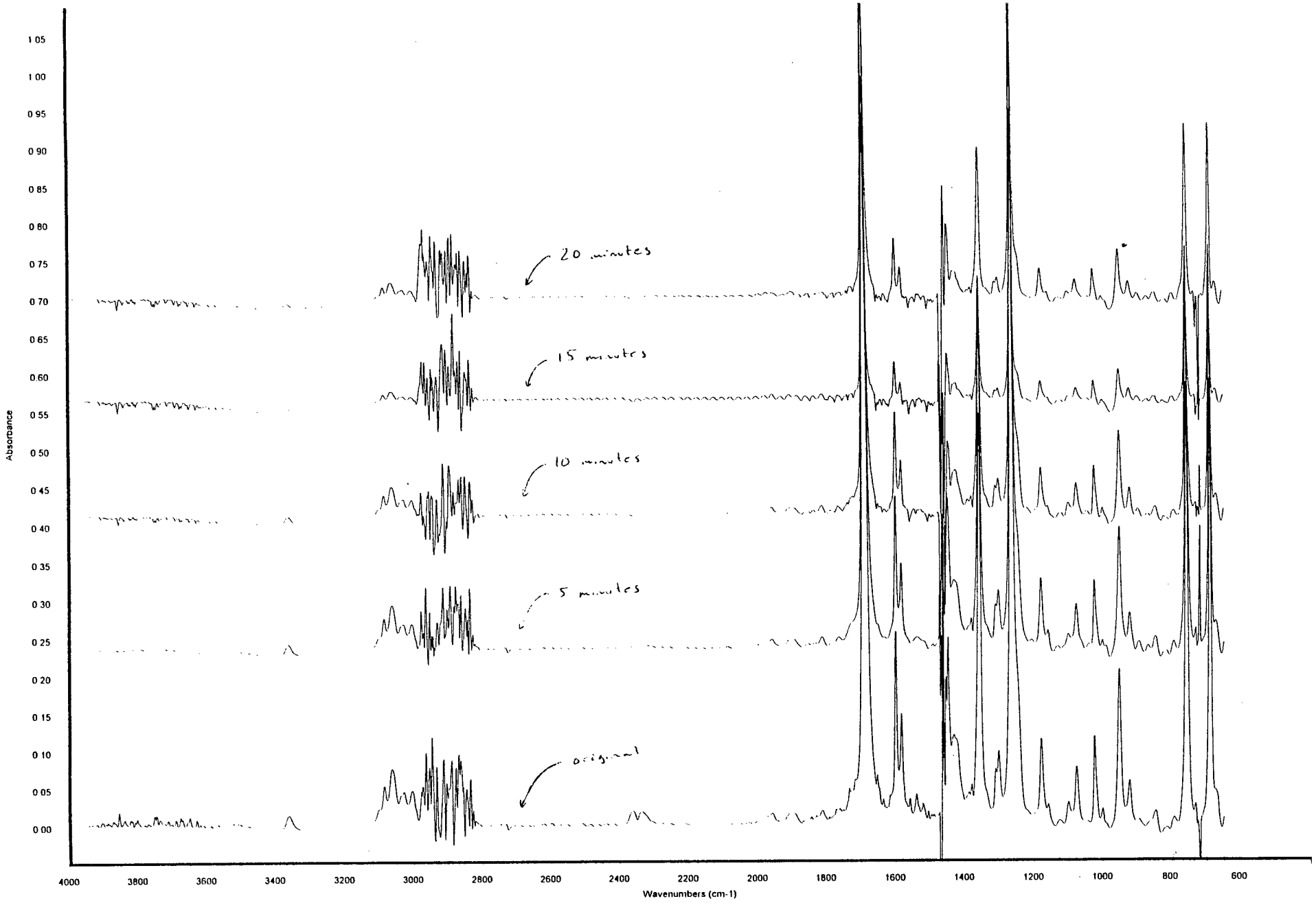
A10

Liquid DMMP


DMMP (Aldrich #D16910-2; CAS:756-79-6; lot#:HF09107DF) by liquid metho
Spectrum Provided by Battelle, trimmed to frequency range of 650-4000 cm-1
Absorbance
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
500
1000
1500
2000
2500
3000
3500
4000
Wavenumber (cm-1)

DMMP on Orange Scotch Tape





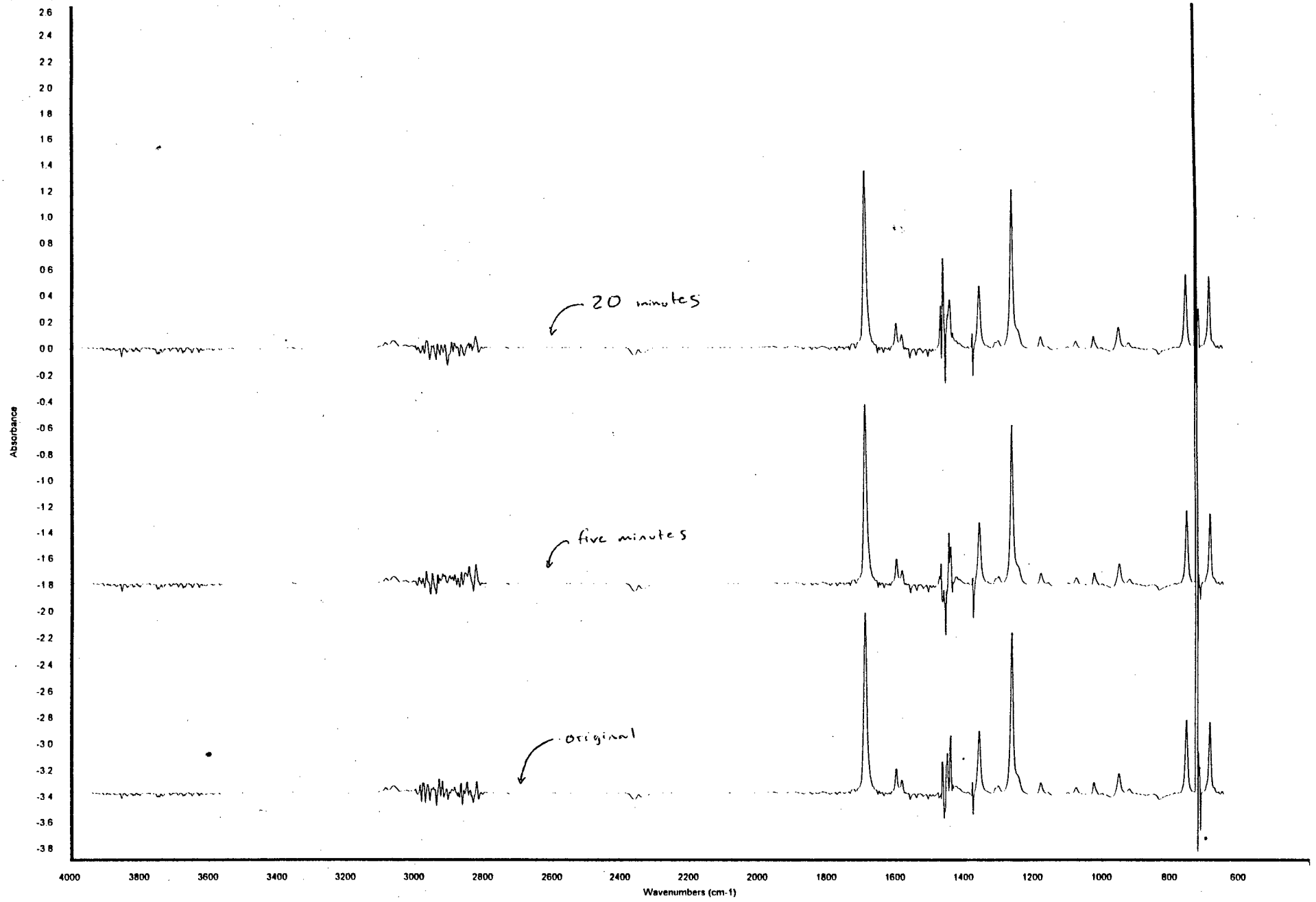
Date Fri Jun 18 10 45 03 1999

Scans 8

Resolution 4 000

Yellow 3M Scotch w/ Acetophenone picked up off beaker

A13



Date: Fri Jun 18 11:41:47 1999

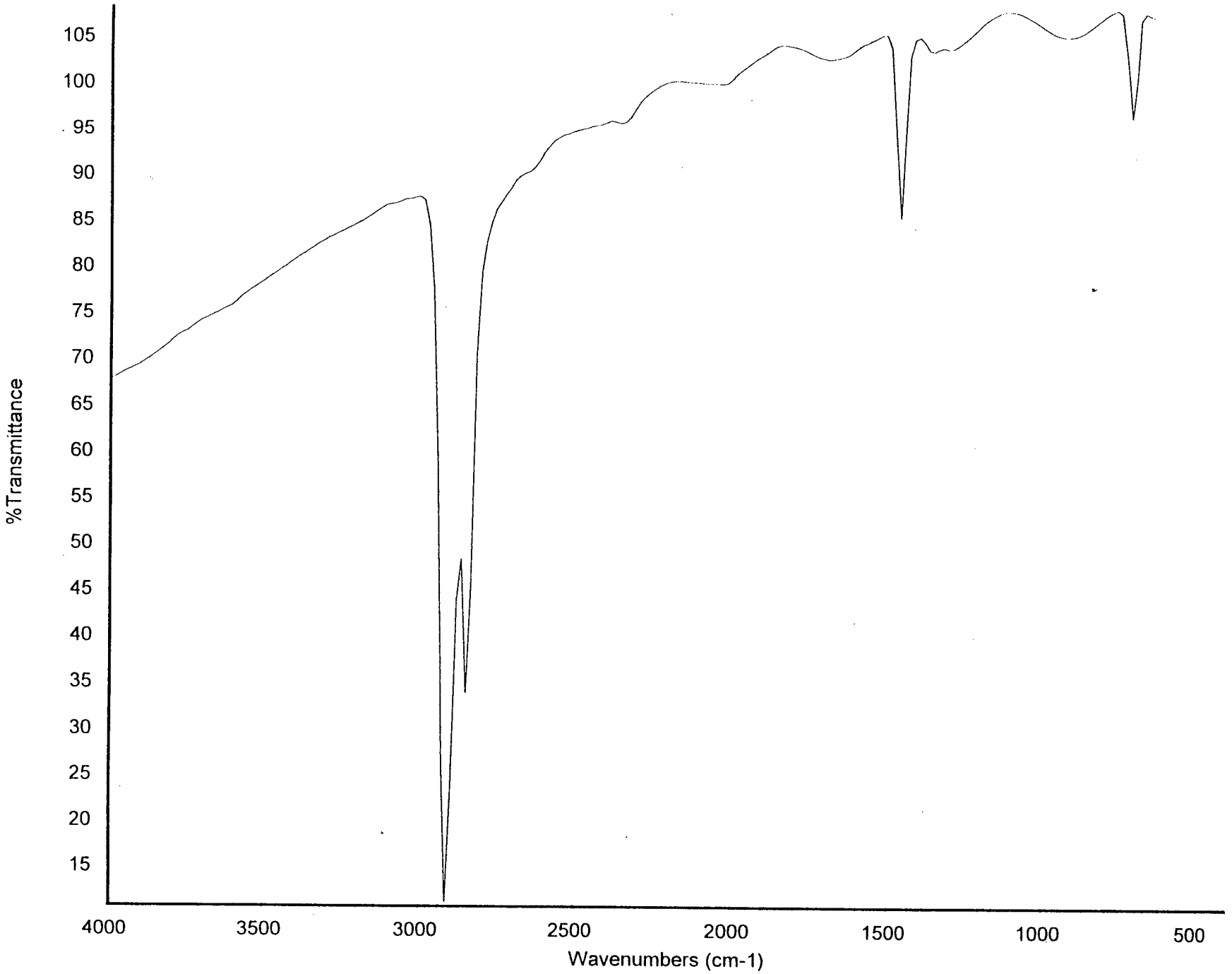
Fri Jun 18 11:41:32 1999

Scans: 8

Resolution: 4.000

Yellow 3M Scotch Tape Double Layered to Preserve
1 mg Acetophenone

A14



Date. Fri May 28 09:45:28 1999

Fri May 28 09:44:52 1999 3-M IR Card KC-0061

Scans: 32

Resolution: 32.000

A15